

Testimony of Dr. Matthews

“As you start to get down to the meat of the issue and you keep your eye focused on what's leading to the adverse events and you do your own calculations, taking this raw data and then redoing it yourself and looking at it this way and looking at it that way, it all keeps stacking back up to the same conclusion”

BEFORE THE AMERICAN ARBITRATION ASSOCIATION
North American Court of Arbitration for Sport Panel

United States Anti-Doping Agency,

Claimant,

v.

Floyd Landis,

Respondent.

**DECLARATION OF SIMON
DAVIS**

AAA No. 30 190 00847 06

I, Simon Davis, declare:

1. I am the Technical Director of MS Solutions. I make this declaration based on my personal knowledge, and if called as a witness, I could and would testify competently to the matters set forth herein.
2. I received a Bachelor of Science Honours in Environmental Biology from Oxford Brookes University in 1991. I received a Ph.D. in Stable Isotope Mass Spectrometry from Liverpool JMU, in association with Cambridge University, in 1996.
3. I was a Stable Isotope Systems Engineer for Micromass UK Ltd. from May 1997 to July 1998. From July 1998 to June 2000, I was a Staff Scientist at the Lawrence Berkley National Laboratory, Berkley University. I rejoined Micromass UK Ltd. as a Development Project Leader for the Inorganic MS Group from June 2000 to March 2003. From March 2003 to March 2005, I was a Research Officer for Queens University. In March 2005, I joined MS Solutions as the Technical Director.

4. Based on my education and experience, I am familiar with the testing commonly used in anti-doping detection: The Testosterone to Epitestosterone ratio test ("T/E test") and the Carbon Isotope Ratio test ("CIR").
5. The T/E test is performed using a Gas Chromatography/Mass Spectrometer ("GC/MS") instrument, which calculates the ratio of testosterone to epitestosterone in a sample. The T/E test calculates this ratio by comparing the specific ion signal – called a "response" – of testosterone to the response of epitestosterone.
6. The GC/MS cannot distinguish between synthetic and natural testosterone, however, because their mass spectra are identical. Epitestosterone is an inert epimer of testosterone, which means that it has the same chemical makeup as testosterone, but because of a difference in chirality (a geometric difference – sort of a mirror image) at one location, epitestosterone is not biologically active.
7. In theory, the ratio of testosterone to epitestosterone in urine in adult males should be approximately 1:1. In fact, however, ratios as high as 15:1 or higher could be normal; conversely, some individuals naturally have low urinary T/E ratios that do not change even with the administration of exogenous testosterone.
8. I am also familiar with the CIR, which also has been described as the test for "synthetic testosterone." Generally, molecules are composed of atoms; specifically, biological molecules are composed primarily of Carbon, Oxygen and Hydrogen atoms. Carbon, in its basic form, is an atom composed of six electrons, six protons and six neutrons. In nature, however, most atoms have one or more stable isotopes. A stable isotope is an atom that has "extra" neutrons. In the case of carbon, ^{13}C is a stable isotope that has one "extra" neutron. The actual carbon isotope makeup of any

individual will vary based on his or her diet. The same is true for plants. As it turns out, the particular plants (mostly soy) used for the building blocks of synthetic or pharmaceutical testosterone are particularly low in ^{13}C , especially when compared to the levels found in most humans.

9. The CIR test is performed using an IRMS instrument, which measures the ratio of $^{13}\text{C}/^{12}\text{C}$ in a target analyte. The theory behind the test is that synthetic testosterone, which is usually soy-based, will be depleted in carbon 13. To account for individual variabilities in things such as diet, which can affect the $^{13}\text{C}/^{12}\text{C}$ ratio, the test compares the $^{13}\text{C}/^{12}\text{C}$ ratio of a testosterone metabolite that is believed to be affected by exogenous testosterone to the $^{13}\text{C}/^{12}\text{C}$ ratio of an endogenous reference compound that is believed not to be affected by exogenous testosterone. By comparing the difference in the $^{13}\text{C}/^{12}\text{C}$ ratio between a testosterone metabolite and a testosterone precursor, or other endogenous reference compound, the CIR test can determine the likelihood of testosterone being from an exogenous source.
10. In theory, for any individual at any one time the $^{13}\text{C}/^{12}\text{C}$ ratio of a testosterone precursor should be identical (or very close) to that of a testosterone metabolite. If a person is using exogenous testosterone, however, there will be a detectable and significant difference between the $^{13}\text{C}/^{12}\text{C}$ ratio in a testosterone metabolite and a testosterone precursor. In other words, if a person is taking exogenous testosterone, his or her $^{13}\text{C}/^{12}\text{C}$ ratio for a testosterone metabolite will be different than the ratio for a testosterone precursor.

11. Once the $^{13}\text{C}/^{12}\text{C}$ ratio, commonly reported in the delta per mil notation ($\delta^{13}\text{C}\text{‰}$), for the testosterone metabolites is calculated, the ratio can be compared with the positivity criteria mandated by the World Anti-Doping Agency.

12. In conjunction with this case, I have reviewed Laboratoire National de Depistage du Dopage's (LNDD) laboratory document package concerning Floyd Landis. I have also reviewed the United States Anti-Doping Agency's responses to Mr. Landis's Second Request for Production.

13. I have relied upon the documents referenced above, my education, my experience, and generally accepted principles within the scientific community in reaching the following conclusions:

- a. The Optima GC 1.67-2 software was originally written for the Micromass Optima IRMS and not the Isoprime IRMS instrument. This software is now 10 years old and can be identified by its code number 1.67-2.
- b. Since version 1.67-2 of software was produced, there have been six major version releases of software for the Isoprime. These include (1) Version 1.67-3 (OS2 operating system) (2) Version 1.67-4 (OS2 operating system), (3) Masslynx Version 3.5i (Windows NT), (4) Masslynx Version 3.6i (Windows NT), (5) Masslynx Version 4.0 (Windows XP) and (6) Ion Vantage Version 1.0 (Windows XP).
- c. The newer versions of the software have the following improvements:
 - i. The newer software includes a new set of electronics with a new set of firmware for the systems head amplifier that corrected errors in the OS2 head amplifier firmware.

the original raw data which can then be compared to those results reported by the LNDD.

14. As to the use and maintenance of a GC-IRMS instrument, I have consulted my copy of the Isoprime User Manual, which I have given to Mr. Landis and his counsel. The Isoprime User Manual states that the operating pressure for the GC-IRMS instrument is between 2 and 4E-6 mbar.
15. I have also reviewed USADA 0176 and confirmed that it shows that the GC-IRMS instrument was operating at a pressure of 5.2E-6.
16. I have relied upon the Isoprime User Manual and USADA 0176, my education, my experience, and generally accepted principles within the scientific community in reaching the following conclusions:
 - a. Operating the GC-IRMS instrument at pressures of 5E-6 millibars or above can result in reduced sensitivity and precision of the reported results and increased variance values. All mass spectrometers require a vacuum in order to operate properly, which involves ensuring that the ion beam can pass from the source to the detector system in a manner consistent with the manufacturer's specifications.
 - b. Failure to operate this machine properly can result in (1) false detection of atmosphere gas as analyte gas and (2) competitive ionization that results in the reduction of the sensitivity of the instrument to analyte gas, among others problems:

- ii. The newer software has the ability to control the GC portion of the GC-C-IRMS, whereas in the OS2 versions of the software, the operator has to manually program the GC.
- iii. The newer software traces any changes that are made to the data post acquisition. For instance, if the software is reprocessed with different integration parameters this would be recorded in all Masslynx and Ion vantage systems, but not in any OS2 systems.
- iv. The newer software contains a standards library, for the automated storage and retrieval of standards values and data. OS2 requires the standards to be applied manually post acquisition.
- v. The newer software has fully documented and tested background subtraction routines. The method and validity of the background routines in the OS2 software is unknown and undocumented. All documentation of the OS2 routines was lost when Micromass purchased Isotech (the developers of the original software).
- vi. The newer software has improved peak detection -- the true nature of the OS2 detection methods is unknown as no documentation remains as to the method used.
- vii. The newer software provides "read-backs" that allow the true state of the Isoprime to be observed and recorded. The OS2 system offers no read backs.
- viii. The newer software works on a modern operating system for which you can obtain up-to-date anti-virus and malware software. OS2 Warp

(the latest software that version 1.67-2 will run on) is no longer supported by IBM and no anti-virus or security software is available.

- ix. The newer software is compatible with a number of Laboratory Integrated Management Systems. This is used for the control of results management.
- d. On account of the age of the software, and the fact that it was not designed for this specific IRMS instrument, there is a serious question about whether it is capable of delivering consistently accurate results.
- e. If the new software were used, it would provide better peak detection, tested and documented background subtraction routines and would remove any errors in the head amplifier firmware. It would also provide a stable and modern operating system with up to date anti-virus and other security software.
- f. Production of the Electronic Data Files ("EDFs") is necessary for the fair adjudication of this matter because the EDFs will assist Mr. Landis' experts in determining whether errors in reporting the results may have stemmed from:
 - i. Errors in the Isoprime head amplifier software.
 - ii. Incorrect and wrongly applied background subtraction.
 - iii. Poor peak integration.
 - iv. Inappropriate reprocessing of the original data files.
- g. Moreover, if the EDFs are provided, it will be possible to identify if the data reported by the laboratory has been reprocessed. Although the OS2 software does not provide a traceable audit of any reprocessing, it is possible to extract

c. Additionally, the increased pressure recorded in the Penning gauge possibly resulted from increased helium pressure in the GC-C system, causing similar defects in results.

d. The increased pressure will result in a decreased lifespan of the source filament, which also may increase the reported measure of variance.

I declare under penalty of perjury under the laws of the State of California that the foregoing is true and correct and that this declaration was executed on February __, 2007 in 13th of February.



Simon Davis

Instructions given to Dr. Botre

From: Sub, Maurice [MSub@gitsondum.com]
Sent: Sunday, April 22, 2007 3:05 PM
To: McLaren, Richard; Francoeo Botre
Cc: Carmen Frobos (E-mail); Chris Campbell (E-mail); Howard Jacobs (E-mail); Ho, James C.; Henry, Janette; Matthew Barnett (E-mail); Patrice M. Brunel (E-mail); Richard R. Young (E-mail); Richard Young's assistant (E-mail); Rosalie Brunel (E-mail); Travis Tygart (E-mail); Davis, Trish
Subject: INSTRUCTIONS IN RE: ELECTRONIC DATA FILES

Follow Up Flag: Follow up
Flag Status: Red

Dear Dr. Botre:

We look forward to your arrival at LMDD tomorrow, Monday, April 23, 2006.

Pursuant to the email of Panel Member Richard McLaren below, we now respectfully provide you with our instructions for handling the Electronic Data Files ("EDFs") in this case.

As you are aware, the EDFs are electronically preserved records of the history of the carbon isotope ratio testing in this case. The carbon isotope ratio testing instruments in this case both allow preservation of electronic records to be analyzed at a future date. They consult the raw data in the case. One of the issues that we face is that the LMDD, in its analysis of Stage 17, used an isotope instrument with (1) very outdated software not designed for this instrument, (2) using wrong specifications, which provided inaccurate results. We believe that properly running the EDFs on current software is an important step in the preparation of the case.

Report on the reprocessing of the Electronic Data Files (EDFs)

**Dr. Francesco Botrè
Independent Expert for the Panel**

Rome, May 10th 2007

USADA 1637

1. Structure of the Report

1.1. This report is formed by a main document of 12 pages and by three Enclosures.

2. Data, documents and information considered in this Report

2.1. The present document is the result of the study of the data obtained following the reprocessing of the electronic datafiles (EDFs) originally generated and processed by the Laboratoire National du Depistage du Dopage (LNDD) in Châtenay-Malabry (Paris, France) on the occasion of the GC-IRMS analysis of the samples "A" and "B" of stage 17. The LNDD is a WADA-accredited and ISO 17025-accredited anti-doping laboratory. GC-IRMS (gas chromatography coupled to isotopic ratio mass spectrometry) is a relatively new technique used by the anti-doping laboratories to discriminate between the endogenous and the synthetic origin of naturally produced steroids, primarily testosterone and its precursors.

2.2. The reprocessing of the EDFs was carried out, under my supervision and responsibility, on May 4th-5th 2007 at the LNDD, at the presence of Technical Experts of both Parties.

2.3. The data to be reprocessed had been previously retrieved, and copied on CD-ROMs, from the LNDD on April 26th 2007, also in this case under my supervision and responsibility and also in this case at the presence of Technical Experts of both Parties.

2.5. The reprocessing of the data was carried out taking into account the instructions reported in the two documents, originally produced by the Representatives of the athlete, that I have received from the Panel (documents transmitted to me in .pdf format on May 2nd 2007, filenames "07-04-22 EDF instructions to Dr. Botre.pdf" and "07-04-29 EDF instructions to Dr. Botre.pdf").

2.6. The documents obtained following the reprocessing of the electronic datafiles (EDFs) have been evaluated also in the light of the information reported on the Laboratory Documentation Packages (LDPs) produced by the LNDD following the original analysis of the "A" and "B" samples.

2.7. Other non-analytical documents, and namely

- the printouts of the list of files/folders present on the computer supports used to produce the CD-ROM containing the EDFs to be reprocessed, and
- the logfile produced by the GC-IRMS system used for the analysis of other 10 (ten) blind "B" samples, and specifically the portion of the file referring to the period April 17th 2007 – April 22nd 2007

have also been considered in this report.

3. Objective of this Report

3.1. The objective of the present document is to report and discuss the data obtained following the reprocessing of the electronic datafiles (EDFs) originally produced and processed on the occasion of the GC-IRMS analysis of the "A" and "B" samples of stage 17.

3.2. The above process imposed also to verify and confirm that the EDFs to be reprocessed were authentic copies of the original files. This involved the study of the backup history of the relevant files and the review of the corresponding "chain of copy".

3.3. Finally, the logfile produced by the instrument used for the GC-IRMS analysis of the 10 additional blind "B" samples, analyzed at the LNDD in the period from April 17th 2007 to April 22nd 2007, was also studied, in order to verify whether other analyses were performed on the same instrument in the same period.

4. Reprocessing of the EDFs: activities carried out at the LNDD on April 26th 2007 and on May 5th-6th 2007

4.1. As outlined above, my involvement as Independent Expert of the Panel included not only the evaluation of experimental data, but also the supervision of all the activities necessary to prepare, and perform, the reprocessing of the EDFs.

4.2. The following activities were therefore carried out under my supervision and responsibility:

- the retrieval (copy) of the electronic data files (EDFs) and of other relevant electronic and printed documents, necessary to perform the reprocessing of the EDFs, carried out at the LNDD on April 26th 2007;
- the actual reprocessing of the EDFs, carried out, again at the LNDD, on May 4th - 5th 2007, and the extraction and printout of the portion of the logfile referring to the period April 17th 2007 - April 22nd 2007, of the new GC-IRMS instrument, also backed up, on a different CD-ROM, on April 26th 2007.

4.3. A record of the above mentioned activities is outlined in two documents:

- "Copy of Electronic Datafiles - Summary of Operation", dated April 26th 2007 (Enclosure #1);
- "Reprocessing of Electronic Datafiles - Châtenay-Malabry (Paris, France) May 4-5 2006 Summary of Operation" (Enclosure #2).

4.4. The above documents were reviewed and undersigned by all present on both occasions. They were also distributed in copy and/or transmitted in electronic format to the Panel and to the Parties.

4.5. For what concerns the description of the activities carried out on both occasions, I entirely confirm the information reported in the Enclosures #1 and #2.

5. Backup history of the EDFs

5.1. Before proceeding with the reprocessing of the EDFs, it has been necessary to review the backup history of the files (the "chain of copy") to be reprocessed.

5.2. This confirmation of the authenticity of the files was deemed necessary since the files obtained on April 26th 2007 had not been copied directly from the internal memory of the instrument, i.e. from the site on which they had been originally stored, but from other CD-ROMs, produced as a part of the back up policy of the LNDD.

5.3. The review of the backup history, and namely the study of the information contained in the printouts obtained on May 5th 2007 and reported at pages 1-36 of Enclosure #3, showed that the chain of copy was documented in its entirety and that it was possible to trace all copies of the original files stored on the internal hard disk of the GC-IRMS instrument.

5.4. Nonetheless, I still considered necessary to further verify the identity of the files. This could be done during the reprocessing of the EDFs on the instrument on which they had been originally created and processed. The evidence obtained is reported later on in this document, at point 7.7.

6. Reprocessing of the EDFs: description of the operation

6.1. As reported in the Enclosure #2, the reprocessing of the EDFs started in the afternoon of May 4th 2007, at 3.45pm, it was suspended at 7.00pm on the same day and it was completed on the following day, May 5th 2007.

6.2. The reprocessing of the EDFs was first performed on the same instrument used for the original analysis of the "A" and "B" samples of stage 17. Once this process was completed, the EDFs were also reprocessed by the new instrument, following their conversion to a readable format, i.e. compatible with the operating system/software installed on the new instrument.

6.3. To carry out the reprocessing of the files on the old instrument it was necessary to copy back the files from the CD to the internal memory of the instrument. Two new folders ("230706" and "040806") were then created on the internal memory of the

instruments, and the relevant files were copied inside these two folders from the CD-ROM produced on April 26th 2007.

6.4. A printout of the list of folders contained in the data directory of the internal hard disk of the instrument was produced immediately before and right after the copying process took place. A further printout of the same kind was produced when the operation was suspended, after deleting the files from the internal hard disk, thus documenting that no EDFs were left on the internal hard disk of the instrument overnight. These three printouts are reported at pages 37-39 of Enclosure #3.

6.5. The reprocessing of the EDFs was carried out, under my supervision and responsibility and at the presence of the Technical Experts of the Parties, by the same analyst of the LNDD who originally processed the data on the occasion respectively of the "A" and "B" analysis, according to the information reported on the Laboratory Documentation Packages.

6.6. However, reprocessing of the EDFs was carried out, both on the old and on the new instrument, without consulting the Laboratory Documentation Packages produced by the LNDD for the analysis of the "A" and the "B" samples. This ensured the analyst to operate in an unbiased way.

6.7. Reprocessing on the old instrument was carried out in three different modes:

- (a) with automatic adjustment of the background
- (b) with manual adjustment of the background
- (c) with no adjustment of the background

Procedures (a) and (c) above are performed automatically by the instrument: the analyst has only to select the relevant option from the menu of the software of the instrument. Procedure (b) is, instead, performed manually by the analyst.

6.8. Three outputs were therefore produced for each EDF reprocessed by the old instrument. Each output consists of two pages, a data page ("Data Processing Results") and a graphical page. The output of each file is then formed by 6 (six) printed pages. These outputs are shown at pages 40-87 (reprocessing of the "A" sample datafiles) and at pages 88-135 (reprocessing of the "B" sample datafiles) of Enclosure #3.

6.9. Additional files that were also reprocessed include the stability runs (for both the "A" and the "B" sample) and the two sets of linearity runs, performed by the LNDD on June 26th 2006 and on July 31st 2006. The data obtained by reprocessing those files are reported at pages 136-149 of Enclosure #3.

6.10. Reprocessing on the new instrument started at 11.20am on May 5th 2007. In this case it was not necessary to first copy the files on the internal hard disk of the instrument, but it was instead necessary to preliminarily convert the EDFs into a format that could be readable by the instrument/software.

6.11. The conversion process could not be performed in a totally automatic way. It was indeed necessary not only to create a new, *ad hoc* folder ("Reprocessing050507"), but also, following the suggestion given by one of the Technical Experts (Dr. Simon Davis), to copy into it some additional files that were

still missing once the automatic conversion process was completed. Practically, these "missing" files were "borrowed" from other data folders containing files obtained by the analysis of other real samples. This process is documented by the printouts reported at pages 185-186 of Enclosure #3. More precisely, page 185 shows the files that are produced after the automatic conversion of the original EDF is completed, while page 186 shows the whole set of files that are necessary to carry out the reprocessing of the file.

6.12. The above process is not described in the manual of the instrument and of the software, nor in the procedure outlined in the document "07-04-29 EDF instructions to Dr. Botre" (see above, point 2.5.). However, following this procedure it was possible to start with the reprocessing of the EDFs.

6.13. Also in this case the EDFs were reprocessed, under my supervision and responsibility and at the presence of the Technical Experts of the Parties, by the same analyst of the LNDD who originally processed the data on the occasion respectively of the "A" and "B" analysis.

6.14. The EDFs were reprocessed following the routine procedure of the laboratory for the analysis of real samples. The processing parameters are shown in the printouts reported at pages 181-182 of Enclosure #3. A representative printout of the plots obtained by this procedure is reported at page 161 of Enclosure #3.

6.15. All those files that had just been reprocessed by the "old" instrument/software were also reprocessed by the new instrument/software. In this case the output of each file is represented only by one printed pages. These outputs are respectively shown at pages 150-160 (reprocessing of the "A" sample datafiles, including the datafiles of the stability runs) at pages 162-174 (reprocessing of the "B" sample datafiles, including the datafiles of the stability runs), and at pages 175-180 (reprocessing of the datafiles of the linearity runs) of Enclosure #3.

6.16. The reprocessing was successfully performed for all EDFs but one, namely the datafile "data010", respective to the fraction 3 of the blank urine analyzed on August 4th 2006 as part of the procedure followed for the "B" sample analysis. This is documented by the screenshots shown at pages 183-184 of Enclosure #3

7. Reprocessing of the EDFs: discussion of the data

7.1. It has to be preliminarily pointed out that the reprocessing of the EDFs on the old instrument has been carried out also following some procedures (i.e. the processing with the totally automatic correction of the background and without any correction of the background) that, even if carried out by the same analyst that performed the original analysis and on the same instruments on which they had been originally generated and processed, are not part of the internal Standard Operating Procedure for GC-IRMS analysis of the LNDD, which instead specifically allows the manual processing.

7.2. This is also true for the procedure of reprocessing the EDFs, after conversion into a readable format, on an instrument/software different from the one by which the same data had been originally generated and processed.

7.3. Nonetheless, I realize that some concern can be due by the fact that the "manual" processing of the analytical signal, especially if not performed correctly, can, in principle, markedly modify the result of the analysis, and that therefore it has to be – whenever possible – clearly ascertained that such a manual process is aimed to improve the quality of the analytical signal, and not to alter the result of the analysis.

7.4. Therefore, all the data obtained by the procedure described in the previous section of this document were evaluated to verify whether some flaws could be discovered in the process of data acquisition and processing originally carried out, on the occasion of the analysis of the "A" and of the "B" samples, by the LNDD.

7.5. As previously reported (6.5), the reprocessing of the files was carried out without referring to the data/plots obtained on the occasion of the original analyses; this means, for instance, that none of the two analysts of the LNDD involved in the reprocessing of the EDFs, nor the responsible of the IRMS Department of the LNDD Dr. Buisson, nor the Director of the Laboratory, Dr. de Ceaurriz, could access the original hardcopies produced for the preparation of the Laboratory Documentation Packages of the "A" and "B" samples, and that no comparison with the data reported on the above mentioned LDPs was ever done by the same people during the reprocessing of the datafiles. This procedure aimed to carry out the process in a way that can be considered equivalent to the one followed by the analysts when they analyze a real sample for the first time.

7.6. It has to be highlighted again that this process is not part of the internal Standard Operating Procedures of the LNDD, and therefore it is not a process covered by the ISO 17025 accreditation nor by the WADA accreditation; more specifically, it also included some procedures – i.e. the processing with the solely automatic correction of the background and without any correction of the background – that are outside of the internal Standard Operating Procedure of the LNDD. This obviously applies also to the analysis of real samples, and not only to the reprocessing of EDFs.

7.7. A first information that was possible to obtain only by re-opening (and not even by reprocessing), on the old instrument, the EDFs, was related to the identity of the files. Based on the filenames, date and time of original acquisition and, in general, on the information available on the data page of the printouts (see previous point 6.8.) there is no evidence that the files are not authentic copies of the original ones, i.e. those originally generated by the instrument on the occasion of the "A" and the "B" analysis.

7.8. The process of conversion of the files from the original format to the new format, performed with the aid of a dedicated software (MassLynx, utility Data Bridge) was not totally automatic, and required some manual adjustment of the operating conditions. It is highly probable that this "manipulation" did not affect at all the real data.

7.9. The final results of the reprocessing of the EDFs, expressed as the difference of the δ values (corrected to take into account the contribution of the acetylation process) between a target analyte (androsterone, etiocholanolone, 5-alpha-diol, 5-beta-diol) and an endogenous reference compound (11-keto-etiocholanolone and pregnanediol) are reported in the tables below, referring respectively to the "A" and the "B" sample.

δ (‰) Difference (corrected values) – sample "A"

	11KE-Andro	11KE-Etio	Prgdiol-5adiol	Prgdiol-5bdiol
Blu Orig	0,48	0,87	1,59	0,55
Blu repr auto	0,49	0,51	3,65	0,92
Blu repr man	0,53	0,56	1,87	0,27
Blu repr noBG	0,02	0,06	1,46	0,47
Blu new instr	0,59	0,09	2,45	1,00
A Orig	3,99	2,58	6,14	2,15
A repr auto	3,14	1,72	5,65	1,70
A repr man	3,65	2,32	6,95	2,65
A repr noBG	2,94	1,87	5,55	2,08
A new instr	3,78	2,18	7,22	2,63

δ (‰) Difference (corrected values) – sample "B"

	11KE-Andro	11KE-Etio	Prgdiol-5adiol	Prgdiol-5bdiol
Blu Orig	0,08	1,08	1,60	0,67
Blu repr auto	0,03	1,11	3,45	1,33
Blu repr man	0,17	0,94	1,89	0,69
Blu repr noBG	0,83	0,25	1,24	0,54
Blu new instr	0,55	0,51	3,66	1,52
B Orig	3,51	2,02	6,39	2,65
B repr auto	1,67	0,32	7,61	3,37
B repr man	1,61	0,35	7,19	3,05
B repr noBG	2,81	1,66	5,58	2,33
B new instr	4,01	2,39	7,03	2,80

Abbreviations: 11KE = 11-keto-etiocholanolone; Andro = androsterone; Etio = etiocholanolone; Prgdiol = Pregnanediol; 5adiol = 5-alpha-diol; 5bdiol = 5-beta-diol; Blu = blank urine; A = sample "A"; B = sample "B"; Orig = original data (as reported on the relevant LDP); repr auto = totally automatic reprocessing, with automatic subtraction of the background; repr man = reprocessing with the manual subtraction of the background and manual integration of the peaks; repr noBG = totally automatic reprocessing, with no subtraction of the background; new instr = reprocessing carried out on the new GC-IRMS instrument.

7.10. The data summarized in the above two tables allow to draw the following observation:

- a) the difference of the δ values between pregnanediol and 5-alpha-diol is always greater than 3, for both the "A" and the "B" sample, regardless the protocol followed to process/reprocess the relevant EDF;
- b) the difference of δ values between pregnanediol and 5-alpha-diol is maximal if the EDFs are reprocessed by the new instrument, both on the "A" and on the "B" samples;
- c) the difference of the δ values of 11-keto-etiocholanolone and etiocholanolone is always smaller than 3, for both the "A" and the "B" sample, regardless the protocol followed to process/reprocess the relevant EDF;
- d) both on the "A" and on the "B" samples, the difference of the δ values of 11-keto-etiocholanolone and etiocholanolone is minimal if the EDFs are reprocessed by the old instrument and by the totally automatic procedure (automatic subtraction of the background);
- e) the difference of the δ values between pregnanediol and 5-beta-diol is always smaller than 3 for the "A" sample, regardless the protocol followed to process/reprocess the relevant EDF; while it is slightly greater than 3 on the "B" sample in the case the EDFs are reprocessed performing either the totally automatic correction of the background or the manual correction of the background;
- f) the difference of the δ values between 11-keto-etiocholanolone and androsterone is slightly smaller than 3 on the "A" sample only in the case the reprocessing is performed automatically and without subtraction of the background; in all other reprocessing modes the difference is greater than 3;
- g) data obtained by the totally automatic procedure (i.e. with the automatic subtraction of the background) gave rise, both on the occasion of the "A" and of the "B" analysis, to a value of the δ difference between pregnanediol and 5-alpha-diol greater than 3 also for the negative reference urine;
- h) data obtained by the reprocessing of the EDFs on the new instrument gave rise, on the occasion of the analysis of the "B" sample, to a value of the δ difference between pregnanediol and 5-alpha-diol greater than 3 also for the negative reference urine.

7.11. The above data also show that the manual subtraction of the background performed by the Paris laboratory, apart from being covered by their internal Standard Operating Procedures, appears to be a scientifically sound process, aimed to improve the quality of the signal and, therefore, the reliability of the obtained results, and not to alter the results of the analysis. This is particularly evident if one considers that the totally automatic reprocessing of the EDFs on the old instrument gave rise to a value of the difference between pregnanediol and 5-alpha-diol greater than 3 also for the negative reference urine, both on the occasion of the "A" and the "B" sample analysis.

7.12. Apart from the numeric data, the appropriateness of the manual subtraction of the background is also evident from the comparison, between the manual and the automatic subtraction of the background, of the baseline of the upper part of the plots reported on the graphical page of the relevant, reprocessed outputs.

7.13. Finally, there was nothing in the data obtained by reprocessing the EDFs related to the stability and to the linearity runs that could invalid the results of the analysis of the "A" and of the "B" sample.

8. Analysis of the 10 "B" blind samples: study of the logfile

8.1. The last set of printouts produced on May 5th 2007 (pages 187-206 of Enclosure #3) refers to the portion of the logfile – referring to the period April 17th 2006 – April 22nd 2006, produced by the GC-IRMS system used for the analysis of the other 10 (ten) blind "B" samples in the same period (see also previous point 2.7.)

8.2. This information had been requested by the Representatives of the Athlete, to verify whether any sample other than the 10 blind "B" samples were assayed in the same period.

8.3. By carefully reviewing the extracted and printed portion of the logfile, I was able to verify that, indeed, it contained information on the analysis of 10 samples only, and of the relevant reference standard, control urine and calibration samples. These samples were identified by the following codes: 1704429, 1804855, 1804423, 1904426, 1904428, 2004856, 2004425, 2104427, 2184865, 2204424. I believe that the first part of the code indicates the date of the analysis, while the second part is an internal identification code of the LNDD.

8.4. The logfile also confirms that three fractions were injected for each one of the 10 samples whose internal codes are reported above. Apparently, only the third fraction of sample code 1904428 was assayed twice, while all other samples were assayed once.

8.5. Not having at hand the information regarding the codes of the 10 blind "B" samples, I am presently unable to verify whether only those samples – and all those samples – have been analyzed in the period April 17th 2007 – April 22nd 2007.

8.6. Nonetheless, this information can be easily verified by comparing the actual, internal laboratory codes of the 10 blind "B" samples with those extracted from the logfile and reported at the point 8.3. above.

8.7. Finally, the time intervals between the single instrumental runs are consistent with the total times of assays, if one considers that the duration of the chromatographic runs is of approximately 45 min if the method applied is the one used for the analysis of (i) all the samples; (ii) the reference urines; and (iii) the acetate calibration mix, and of approximately 15 min if the method applied is the one used for the analysis of the alkane calibration mix.

9. Summary of the Conclusions

9.1. The review of the backup history of the EDFs, and their reprocessing on the same instrument originally used for the analysis of the "A" and the "B" samples of stage 17 did not show anything that can indicate that those files are not an authentic copy of the original files. Therefore, the EDFs retrieved on April 26th 2007 and reprocessed on May 4th-5th 2007 can be considered authentic copies of the corresponding original data files, i.e. of those electronic files produced, under the OS-2 operating system, by the GC-IRMS instrument used on the occasion of the analytical operations carried on the "A" and "B" sample of stage 17, respectively on July 23rd 2006 and on August 4th 2006.

9.2. The reprocessing of the EDFs on the same instrument originally used for the analysis of the "A" and the "B" samples of stage 17, carried out in three different processing modes (with automatic correction of the background, manual correction of the background and with no correction of the background) showed that:

- filenames, date and time of acquisition are identical to those reported on the Laboratory Documentation Packages of the "A" and "B" sample, thus further confirming that the EDFs are authentic copies of the originals;
- the manual correction of the background – originally carried out on the occasion of the analysis of the "A" and "B" samples – had been carried out correctly and appropriately, this meaning (i) that this procedure had been performed in agreement with, and without any deviation from, the internal Standard Operating Procedures of the laboratory, and (ii) that it was aimed, and indeed allowed, to improve the quality of the instrumental signal;
- the reprocessed data, regardless the variability of the individual results, show that, in all cases, the difference of the δ values between pregnanediol and 5-alpha-diol is greater than 3, for both the "A" and "B" samples, also taking into account the stated value of the measurement uncertainty value (0.8‰).

9.3. The conversion of the original datafiles into a format that could be processed by the new instrument and by the new software was a process that could not be performed in a totally automatic way. It indeed required some extra activities not described in the procedure reported on the manual of the instrument/software and not included in the documents, transmitted to me in electronic format, "07-04-22 EDF instructions to Dr. Botre.pdf" and "07-04-29 EDF instructions to Dr. Botre.pdf".

9.4. However, giving for granted that the conversion of the original datafiles into a format that could be processed by the new instrument and software was completely successful, it can be stated that the results obtained after the reprocessing of the datafiles on the new instrument – which is an almost completely automatic process – led to the same final result (i.e. a difference of the δ values between pregnanediol and 5-alpha-diol greater than 3, on both the "A" and "B" sample, also taking into account the stated measurement uncertainty) obtained by both the original manual process and by reprocessing the data on the old instrument with different process parameters.

9.5. Finally, the study of the logfile, and specifically of the portion that was extracted and printed out on May 5th 2007, confirmed that only 10 samples, identified by the internal laboratory codes reported at point 8.3 of the present document, were analyzed by the new GC-IRMS instrument in the period from April 17th to April 22nd 2007. The relevant portion of the logfile indeed shows that only those 10 samples, together with the relevant reference standards, calibration samples and control urines, were analyzed in the above mentioned period. It would be very simple to verify whether those 10 codes actually refer to the codes of the 10 blind "B" samples. The study of the logfile also confirmed that the time intervals between the single instrumental runs are consistent with the actual times of the GC-IRMS assays.



Dr. Francesco Botrè

Attachment #1

COPY OF ELECTRONIC DATAFILES SUMMARY OF OPERATION

On April 26th 2007, at 1.00 pm, at the Laboratoire National du Depistage du Dopage (LNDD) in Chateaufort, representatives of Mr Landis and of the USADA have met at the presence of an independent expert of the Panel to carry out the retrieval of the Electronic Data Files (EDFs).

The following people were present:

Dr. Simon Davis, Dr. Will Price (scientific experts of Mr. Landis), Dr. Larry Bowers, Dr. Jeanine Jumeau (scientific experts of the USADA), Dr. Jacques de Ceauriz (Director of the LNDD), Dr. Corinne Buisson (Responsible for the IRMS Department of the LNDD) Dr. Francesco Botrè (Independent expert for the Panel)

Prior to begin the extraction of the EDFs, the Parties discussed on which were the files to be extracted, whether only the files of the sample (A and B) collected on the occasion of the stage 17, or also those referring to the 10 blind "B" samples analyzed by IRMS in the period from April 17th to April 22nd 2007. Dr. Botrè called by phone Prof. Richard McLaren, who, on behalf of the panel, communicated to retrieve also the data of the 10 blind samples, but to store them on a separate support.

Due to the size and features of the files to be extracted, it was decided to use CD-ROMs as the electronic support to be used for the back up.

It was agreed that only one copy of the data would have been produced and that this copy will be given in custody to Dr. Botrè.

The datafiles of the IRMS analysis of the "A" and "B" sample of stage 17 were not copied directly from the hard disk of the instrument, but from a CD-ROM on which they had been previously backed up, as part of the internal procedures of the laboratory, by the personnel of the LNDD. This transfer was made by removing the internal hard disk of the instrument, connecting it to a PC with a CD-writer and then re-installing the hard disk back in place inside the instrument.

During the extraction of EDFs of the 10 blind "B" samples three further issues came out:

- 1) Dr. Davis requested that not only the datafiles, but also the logfiles, had to be extracted and backed up
- 2) Dr. Buisson explained that one calibration file (filename: 1704mixcalIRMS01.raw) did not match the criteria of the laboratory and therefore it was decided not to use it; this is the reason why four calibration files, and not three, were saved on the memory of the computer
- 3) The file named "1704bruit1.raw" refers to an instrumental check for the background noise

The following decisions were taken:

- 1) It was decided to copy also the logfiles, but to store them on a separate CD-ROM.
- 2) The file named "1704mixcalIRMS01.raw" was also copied, on the same CD containing the datafiles of the 10 blind "B" samples.
- 3) The file named "1704bruit1.raw" was also copied as above

A total of 3 CD were therefore produced. The CD were labelled as follows

"Electronic Data Files Stage 17 A+B + Linearity"

"B Sample analysis 10 Blind Samples"

"Logfile"

The content of the CD (Folders/Subfolders) is reported in the Attachment #1.

The 3 CDs were placed in sealed envelopes signed by Dr. Davis, Dr. Bowers Dr. de Ceaurriz, Dr. Buisson and Dr. Botrè.

At the end of the backup process, the Parties discussed on how to proceed for the reprocessing of the datafiles. Dr. Thomas Brenna (USADA expert) also participated to this discussion by phone. It was agreed that Mr. Landis representatives and experts will prepare a detailed request to the Panel, outlining not only the reasons why they think the reprocessing is important, but also the technical details of the procedure.

The operations ended at 7.30pm.

Chatenay-Malabry, April 26th 2007.

Dr. Simon Davis

Dr. Will Price

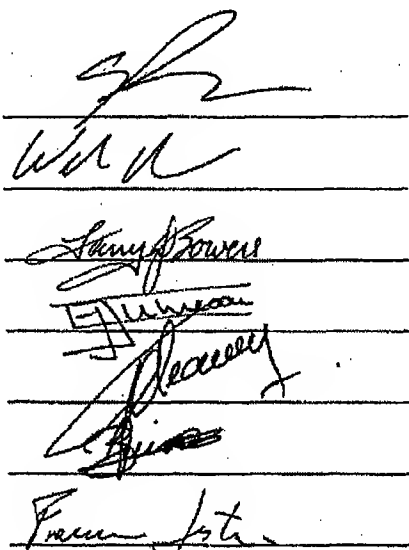
Dr. Larry Bowers

Dr. Jeanine Jumeau

Dr. Jacques de Ceaurriz

Dr. Corinne Buisson

Dr. Francesco Botrè



Attachment #2

REPROCESSING OF ELECTRONIC DATAFILES
Chatenay-Malabry (Paris, France) May 4-5 2006
Summary of Operation

On May 4th 2007, at 1.00 pm, technical experts of Mr Landis and of the USADA have met in the presence of an independent expert of the Panel at the Laboratoire National du Dopage (LNDD) in Chatenay Malabry, to carry out the reprocessing of the Electronic Data Files (EDFs) copied on April 26th 2007.

The following people were present:

Dr. Simon Davis (scientific expert of Mr. Landis), Dr. Thomas Brenna, Dr. Jeanne Jumeau (scientific experts of the USADA), Dr. Jacques de Ceaurriz (Director of the LNDD), Dr. Corinne Buisson (Responsible for the IRMS Department of the LNDD) Dr. Francesco Botrè (Independent expert for the Panel).

Before proceeding with the re-processing of the EDFs, Drs. de Ceaurriz and Buisson were asked to supply additional information on the process of backup of electronic data, either in general and with specific reference to those data copied on the three CD-ROMs on April 26th 2006 and still under the custody of the independent expert of the Panel. This mainly to further clarify the meaning of the following statement, reported at page 1 of the document "Copy of Electronic Datafiles - Summary of Operation" dated April 26th 2007:

"The datafiles of the IRMS analysis of the "A" and "B" sample of stage 17 were not copied directly from the hard disk of the instrument, but from a CD-ROM on which they had been previously backed up, as part of the internal procedures of the laboratory, by the personnel of the LNDD."

The following information was obtained:

- The process of backup of the datafiles from the internal memory of the old Isoprime (S.N. JA 010), i.e. the instrument on which the instrumental analysis of the "A" and "B" samples of stage 17 was performed, is periodically carried out manually, under the responsibility of the LNDD. In practice, these operations are performed by an external company that has a specific contract with the LNDD.
- Specifically, the original backup of the data concerning the analysis of the "A" and "B" samples of stage 17 was carried out on October 31th 2006. This is the process that was performed by removing the internal hard disk of the instrument, connecting it to a PC with a CD-writer and then re-installing the hard disk back in place inside the instrument. The data were stored on two CD-ROMs, labelled as "Backup du 31/10/2006 Data: 010206 → 251006 CD1" and "Backup du 31/10/2006 Data: 010206 → 251006 CD1". These are to be considered the two "master" CDs.
- The data stored on the two above mentioned master CDs were then copied, for practical reasons (i.e. to allow a faster retrieval of information), on more, additional CDs. Particularly, the data concerning the analysis of the "A" sample of stage 17 had been archived, together with other data files, also into another CD, labelled as "Isoprime I Data Juin 06 à 31/07/06 (créé le 30/01/07)".
- Finally, the data concerning the analysis of the "A" and "B" samples of stage 17 were also copied on another CD, labelled as "Isop I 23/07/06 04/08/06"; this last CD, produced in the morning of April 26th 2007, contained only the data of the

two samples without any additional data (e.g. those of the assays to assess the linearity).

- The CD labelled as "Electronic Data Files Stage 17 A+B + Linearity", produced in the afternoon of April 26th 2007 at the presence of the technical experts of the Parties and of the independent expert of the Panel, contained copies of files stored on both the CD labelled as "Isop 1 23/07/06 04/08/06" (data files of samples "A" and "B" of stage 17) and on the CD labelled as "Isoprime I Data Juin 06 à 31/07/06 (créé le 30/01/07)" (data files of the linearity assays).

The above back-up history was documented by obtaining hardcopies of the list of folders/files stored on each one of the above mentioned CD-ROMs.

Prior to begin the extraction of the EDFs, the Parties discussed on which were the files to be reprocessed, whether only the files of the sample (A and B) collected on the occasion of the stage 17, or also those referring to the 10 blind "B" samples analyzed by GC-IRMS in the period from April 17th to April 22nd 2007. The discussion also involved the request of Mr. Landis' expert to access the logfile copied on the CD-ROM labeled as "Logfile" on the occasion of the previous meeting at the LNDD, on April 26th 2006. According to the instructions received by the President of the Panel, Mr. Patrice Brunet, in his email message of May 3rd 2007, Dr. Botrè tried without success to contact by phone Mr. Richard Campbell, and left a message on his mobile phone voicemail. In order to optimize the timeframe of operation, it was agreed to go on anyway with the reprocessing of the EDFs of the "A" and "B" samples from stage 17 first, and to go back to the issue of the 10 blind samples datafiles and of the logfile later on.

The sealed envelope containing the CD-ROM labelled as "Electronic Data Files Stage 17 A+B + Linearity" was then opened by Dr. Botrè and, at 3.05 pm, all present moved from the meeting room to the laboratory room hosting the GC-IRMS instruments.

The reprocessing of the EDFs was first performed on the same instrument used for the original analysis of the "A" and "B" samples of stage 17. The process was performed in the presence of Drs. Davis, Brenna, Juneau, Buisson and Botrè, while Dr. de Ceauriz was not constantly present in the GC-IRMS room.

To carry out the reprocessing of the files it was necessary to copy back the files from the CD to the internal memory of the instrument. It was indeed not possible to load and reprocess the data directly from the CD-ROM. Two new folders ("230706" and "040806") were then created on the internal memory of the instruments, and the relevant files were copied inside these two folders from the CD-ROM. A printout of the list of folders contained in the data directory of the internal hard disk of the instrument was produced immediately before and right after the copying process took place.

Once the files were copied, it was possible to start with their reprocessing. It was agreed that an analyst of the LNDD (namely, the same person who originally processed the data on the occasion respectively of the "A" and "B" analysis, according to the information reported on the Laboratory Documentation Packages) would have operated the computer to carry out the reprocessing of the datafiles, under the responsibility and at the presence of the independent expert of the Panel and at the presence of the experts of the Parties.

It was agreed that three outputs had to be produced for each file: (a) a first one, applying the automatic subtraction of the background; (b) a second one, manually

subtracting the background; (c) a third one, with no background subtraction. Each output consists of two pages, a data page ("Data Processing Results") and a graphical page. The output of each file is then represented by 6 (six) printed pages.

It was agreed to start with the reprocessing of the datafiles of the "A" sample, i.e. with those copied in the folder "230706".

The reprocessing of the files started at 3.45pm. The reprocessing of the datafiles of the "A" sample was completed without comparing the outputs with those reported in the Laboratory Documentation Packages.

At 7.00pm, it was agreed to suspend the operation to continue on the following day.

It was agreed to delete the files (both those just reprocessed and those still to be reprocessed) copied on the internal hard disk of the instrument. A printout of the new list of folders in the data directory was also produced.

The operations ended at 7.30pm. It was agreed to continue the operation on the following day, May 5th, at 9.00am.

The operations started again at 9.15am on May 5th 2007. In a preliminary meeting, it was agreed to complete the process suspended the day before. All the presents moved again to the laboratory room hosting the GC-IRMS instruments at 9.30am. The files not yet reprocessed were copied again on the internal hard disk of the instrument.

The above files were then reprocessed, starting with the EDFs of sample "B", then proceeding with the two sets of the stability runs (one for the "B" and one for the "A" sample), and with the two sets of linearity runs originally performed on June 26th 2006 and on July 31st 2006. Also in this case the reprocessing of the datafiles was completed without comparing the outputs with those reported in the Laboratory Documentation Packages.

After all the above processes were completed, the data were deleted again from the internal hard disk of the instrument.

At 11.20am it was agreed to start the reprocessing of the EDFs on the new instrument. The conversion of the files was carried out by Dr. Buisson, under the supervision of the independent expert of the Panel and at the presence of the technical experts of the parties. A new directory ("Reprocessing050507") was created on the internal hard disk of the instrument and the procedure outlined in the document "07-04-29 EDF Instructions to Dr. Botrè" was followed. The files were successfully converted and saved into the new directory; nonetheless, it was not possible to reprocess them. Dr. Davis explained that this could be due to the lack of some "support" files that are automatically generated during the analysis of real samples and that, consequently, were not present in the new directory. To proceed with the reprocessing of the datafiles, it was agreed to copy, from other data directories into the newly created directory, also those support files. This procedure allowed to open the files under the software of the instrument and to reprocess them for the first time by this instrument.

It was agreed to reprocess all those files that had just been reprocessed by the original instrument. The reprocessing of the files was carried out by the same two analysts who had just reprocessed the data on the original instrument.

The files were reprocessed following the routine procedure of the laboratory for the analysis of real samples. Two printouts were produced to show the routine processing parameters of the laboratory, which were applied also for the reprocessing of the datafiles.

It was possible to complete the reprocessing of all files, apart from the datafile "data010" (respective to the blank urine fraction 3 of the "B" analysis), for which an error message appeared. This is documented by two printouts of the screenshot.

At 1.45pm the reprocessing of the datafiles on the new instrument was completed. All the presents left the GC-IRMS laboratory and went to the meeting room.

At this point, Dr. Davis stated that the reprocessing of the datafiles of the 10 blind samples was no longer necessary. It was therefore agreed not to open the sealed envelope containing the corresponding CD-ROM, which remained under the custody of the independent observer. It was no longer necessary to contact the Panel for this issue.

With respect to the issue of the logfile - copied on the CD-ROM labelled as "Logfile" on April 26th 2007 - Dr. Botrè felt it necessary to ask for the opinion of the Panel, also to decide whether or not to supply copies of the printouts produced during the 2-days operation to the Parties. To this respect, Dr. Botrè repeatedly tried to contact - sequentially - Mr. Campbell, Mr. McLaren and Mr. Brunet, without success. At around 15.35pm Mr. McLaren called back. The following indications were supplied to Dr. Botrè: (1) the Parties could be given copies of the printouts, provided it was possible to clearly mark all the copies as originals; (2) it was also agreed that it was not possible to printout the content of the logfile and to give it to the Parties its entirety, since it contained confidential information regarding the activity of the lab for samples other than Mr. Landis'. It was agreed to cut-and-copy only the portion regarding the six days (April 17-22) in which the analysis of the 10 blind samples took place.

A total of 206 printed pages (numbered from 1 to 206) were produced during the two-days of operation. The original copy is under the custody of Dr. Botrè. The other three copies, with every page stamped in red as "Copie certifiée conforme des données et formulaires originaux", were distributed to the Parties and to the LNDD.

The operation ended at 5.20 pm.
Chatenay-Malabry, May 5th 2007.

Dr. Simon Davis

Dr. Thomas Brenna

Dr. Jeanine Jumeau

Dr. Jacques de Ceaurriz

Dr. Corinne Buisson

Dr. Francesco Botrè

995474	Original Result	Auto	Manual	Zero	Masslynx
A Sample					
E-11K	-2.58	-1.72	-2.32	-1.76	-2.18
A-11K	-3.99	-3.14	-3.65	-2.94	-3.78
5B-P	-2.15	-1.70	-2.65	-2.08	-2.63
5A-P	-6.14	-5.65	-6.96	-5.55	-7.22
B Sample					
E-11K	-2.02	-0.32	-0.35	-1.66	-2.39
A-11K	-3.51	-1.67	-1.61	-2.81	-4.01
5B-P	-2.65	-3.37	-3.05	-2.33	-2.80
5A-P	-6.39	-7.61	-7.19	-5.58	-7.03

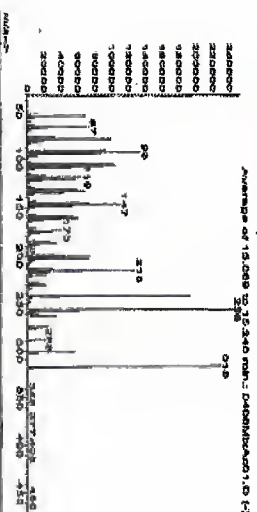
Blanks					
A Sample					
E-11K	-0.87	-0.51	-0.56	-0.06	0.09
A-11K	-0.48	-0.49	-0.53	-0.02	-0.59
5B-P	-0.55	-0.92	-0.27	-0.47	-1.00
5A-P	-1.59	-3.65	-1.87	-1.46	-2.45
B Sample					
E-11K	-1.08	-1.11	-0.94	-0.25	-0.51
A-11K	-0.08	0.03	0.17	0.83	0.55
5B-P	-0.67	-1.33	-0.69	-0.54	-1.52
5A-P	-1.60	-3.45	-1.89	-1.24	-3.66

Peak Identification – GC/MS

Mass Spectra Full Scan

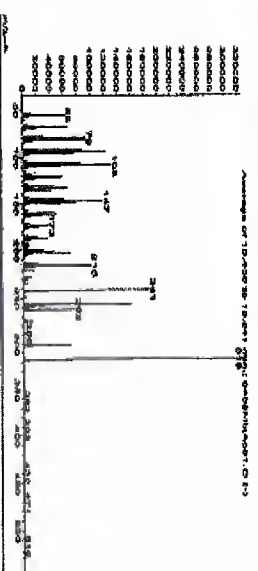
- Fraction 3 : 36 undecene-3e,17b-diol diacetate

- Mix spectra



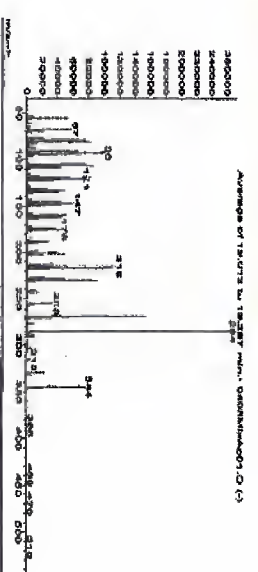
- Fraction 3 : 3e undecene-3e,17b-diol diacetate

- Mix spectra

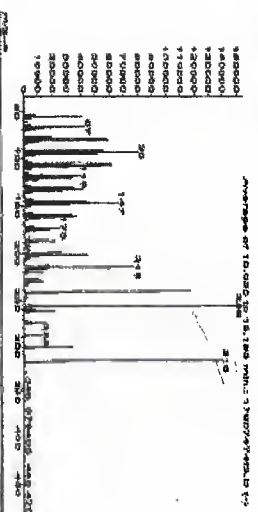


- Fraction 3 : 53 propano-3e,20e-diol diacetate

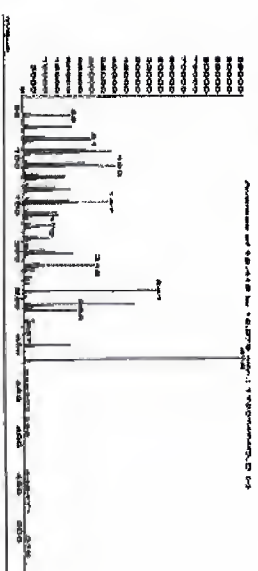
- Mix spectra



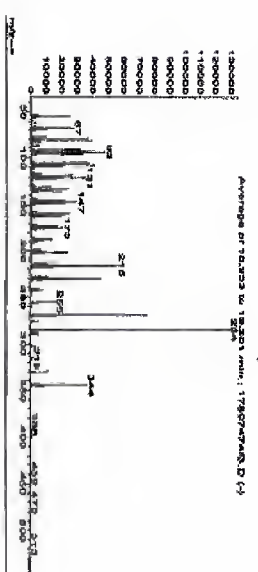
- Sample 17807 0954748



- Sample 17807 0954748



- Sample 17807 0954748



Full Scan of Peaks in Fraction 3
shows that peaks are pure and properly identified

GC/MS Retention Times Match and are Recorded

Substance caractérisée :

3a Androstane-3a,17b-diol diAC

Fichier :

17807474E

Mél					Echantillon				
Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)
15.19	1.420	100	90.8	76.4	15.14	1.418	100	91.0	76.9
Tolérance basse	13.04	1.406	80.8	66.4					
Tolérance haute	13.34	1.434	100.8	86.4					

Concordance des Tr :

oui

X

non

Concordance des Tr :

oui

X

non

Concordance globale des abondances relatives :

oui

X

non

Substance caractérisée :

3a Androstane-3a,17b-diol diAC

Fichier :

17807474E

Mél					Echantillon				
Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)
15.58	1.457	100	57.8	49.5	15.50	1.452	100	59.6	49.9
Tolérance basse	13.42		47.8	39.6					
Tolérance haute	15.74		67.8	59.4					

Concordance des Tr :

oui

X

non

Concordance des Tr :

oui

X

non

Concordance globale des abondances relatives :

oui

X

non

Substance caractérisée :

3a Androstane-3a,17b-diol diAC

Fichier :

17807474E

Mél					Echantillon				
Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Tr	M1 (%)	M2 (%)	M3 (%)
19.21	1.795	100	56.3	28.0	19.12	1.791	100	56.1	27.3
Tolérance basse	19.02		46.3	22.4					
Tolérance haute	19.40		66.3	33.6					

Concordance des Tr :

oui

X

non

Concordance des Tr :

oui

X

non

Concordance globale des abondances relatives :

oui

X

non

USADA 0324

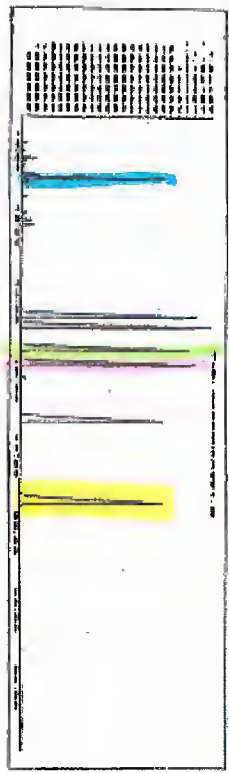
45

氏名	年齢	性別	職業	備考
佐藤 一郎	25	男	学生	
田中 美子	22	女	主婦	
山田 健二	28	男	会社員	
鈴木 花子	20	女	学生	
高橋 隆夫	30	男	会社員	
中村 由美	23	女	主婦	
小林 大輔	27	男	会社員	
渡辺 真理子	21	女	学生	
森田 浩二	29	男	会社員	
伊藤 千恵	24	女	主婦	
清水 健一	26	男	会社員	
山口 美穂	22	女	学生	
佐々木 隆夫	31	男	会社員	
木村 由美子	23	女	主婦	
高木 大輔	27	男	会社員	
中村 真理子	21	女	学生	
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渡辺 真理子	21	女	学生	
森田 浩二	29	男	会社員	



Peak Identification – GC/MS Order of Elution and Identification under WADA TD2003IDCR

Mix Acetate (all peaks of interest) - Ex. 25, USADA0309



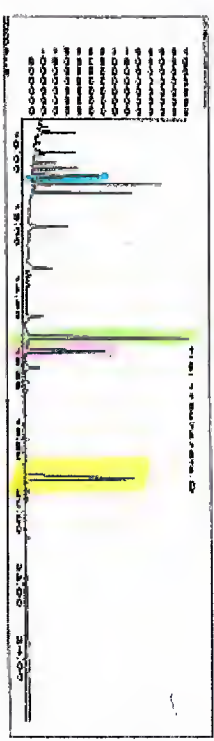
Nom	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstano AC	14.37	1.343	258	14,558,348
Etiocholanone AC	14.53	1.369	272	27,927,453
Androstano AC	15.18	1.420	256	46,140,764
5b Androstano 3a 17b diol d4AC	15.58	1.457	316	27,022,036
5a Androstano 3a 17b diol d4AC	17.09	1.597	271	37,046,436
11 Ketotestosterone AC	19.21	1.738	264	36,707,461
5b Progestano 3a 20a diol d4AC	29.84	4.052	254	46,052,026

Temps de rétention, temps de rétention relatif et target Signal (M1)



Nom	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstano AC	0.00	0.000	258	5,912,258
Etiocholanone AC	0.00	0.000	272	0
Androstano AC	16.19	1.621	256	30,661,343
5b Androstano 3a 17b diol d4AC	16.84	1.683	316	9,191,814
5a Androstano 3a 17b diol d4AC	0.00	0.000	271	0
11 Ketotestosterone AC	19.17	1.793	264	29,874,793
5b Progestano 3a 20a diol d4AC	29.84	2.984	254	29,874,793

Temps de rétention, temps de rétention relatif et target Signal (M1)



Nom	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstano AC	0.00	0.000	258	4,350,522
Etiocholanone AC	0.00	0.000	272	0
Androstano AC	15.14	1.514	256	15,590,188
5b Androstano 3a 17b diol d4AC	15.50	1.550	316	9,733,825
5a Androstano 3a 17b diol d4AC	0.00	0.000	271	0
11 Ketotestosterone AC	19.17	1.791	264	20,380,359
5b Progestano 3a 20a diol d4AC	29.84	2.984	254	20,380,359

Temps de rétention, temps de rétention relatif et target Signal (M1)

Blank Urine F3
Ex. 25, USADA0319

Sample 995474 F3
Ex. 25, USADA0321

GC/C/IRMS Retention Times Recorded and Matched

LNDD	ENREGISTREMENT	Codification : F-PCR-06
		Version : E Date : 24/11/05 Page : 1/2
FICHE D'ANALYSE / RESULTATS GC/CIRMS		

Echantillon :	17807 B995474	Instrument :	GC/CIRMS Isoprime 1
Répartiteur :	040806	CO et graphique :	2603

Fructose F3 (Dish)

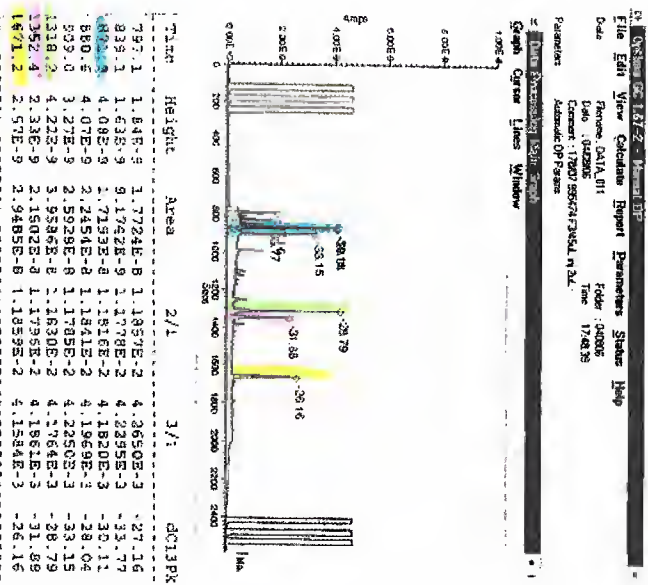
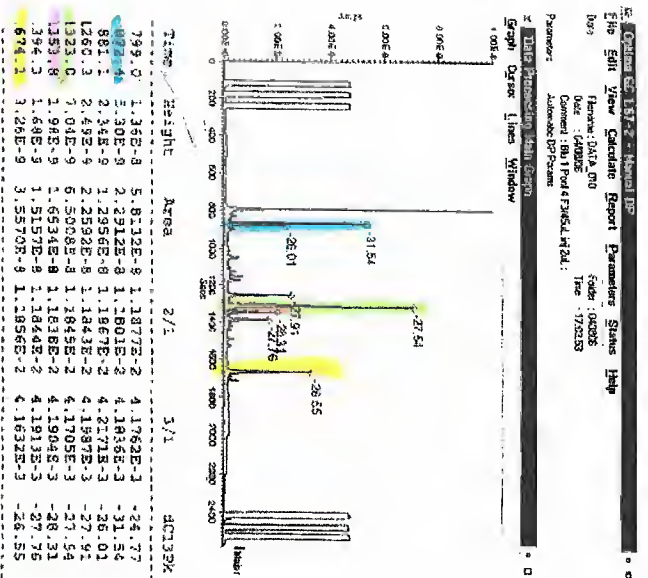
Bilan de l'analyse				
SI	SB Admis	Sc Admis	SB Périod	
Nom du fichier	data_010	data_010	data_010	
α (°)	8.71	1323	1334	1674
αr	-	1517	1552	1919
Intensité (a.u.)	53	7.0	2.0	3.3
δ ¹³ C Su. mesurée	-31.34	-27.54	-28.31	-26.55
δ ¹³ C Su. corrigée	-	-22.18	-23.11	-21.31

Références				
SI	SB Admis	Sc Admis	SB Périod	
Nom du fichier	data_011	data_011	data_011	
α (°)	8.72	1318	1352	1671
αr	-	1512	1553	1917
Intensité (a.u.)	4.1	4.2	2.3	2.6
δ ¹³ C Su. mesurée	-30.11	-28.79	-31.88	-26.16
δ ¹³ C Su. corrigée	-	-23.49	-27.41	-21.05

GC/CIRMS: Retention Times Match

Blank Urine F3, Ex. 25, USADA346-347

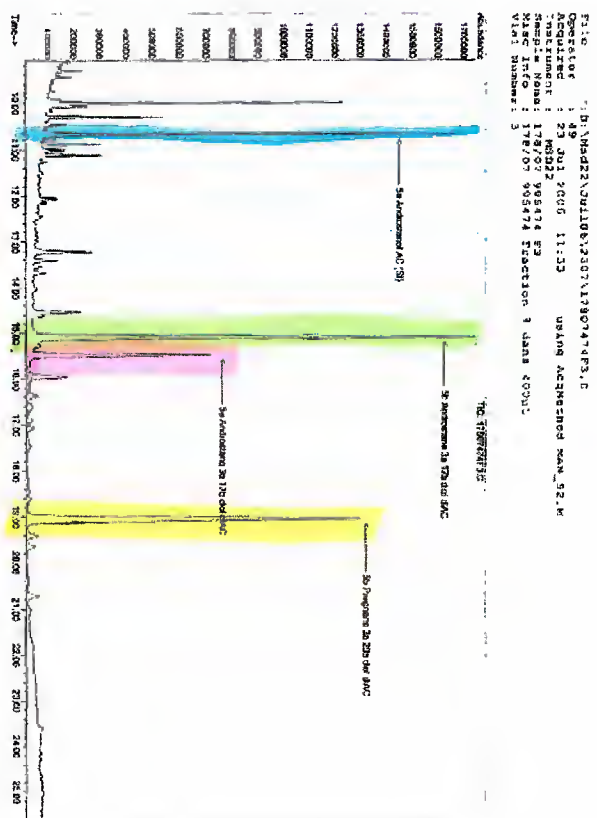
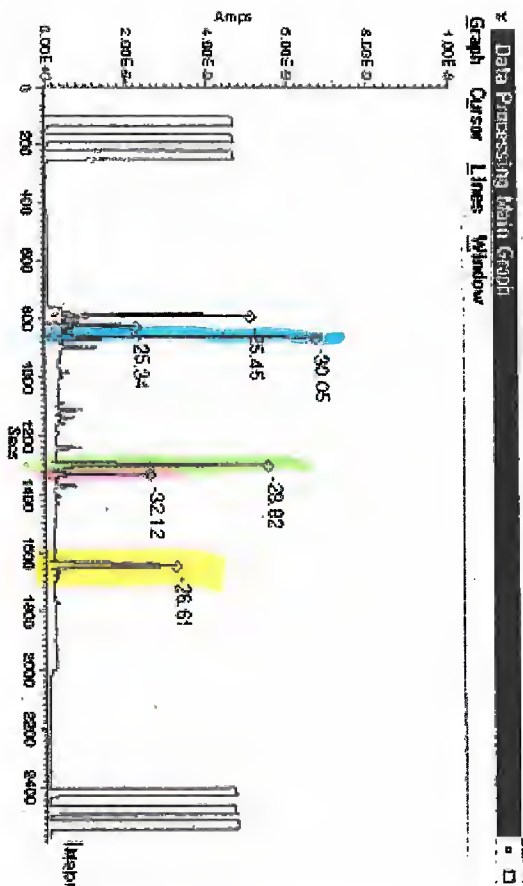
Sample 995474 F3, Ex. 25, USADA349-350



Mix Cal Acetate, Ex. 25, USADA360

Time	Height	Area	2/1	3/1	GC13PK
4.30E-9	4.30E-9	2.0644E-8	1.1855E-2	4.1628E-1	-30.40
12.41.8	4.35E-9	1.5212E-8	1.1934E-2	4.1653E-1	-31.92
13.16.7	3.35E-9	2.6894E-8	1.1777E-2	4.1663E-1	-33.77
14.31.1	2.83E-9	3.1504E-8	1.1971E-2	4.1687E-1	-35.76

GC/CIRMS Matches GC/MS A Sample



Temps de rétention, temps de référence relatif et temps Standard (min)

Nom	Ret Time	Ref Time	Temp Standard	Temp Reference
50 Acetone AC	0.00	0.000	256	2,492.728
50 Acetone AC	0.00	0.000	272	0
50 Acetone AC	15.10	1.415	256	3,658.105
50 Acetone AC	15.48	1.480	256	1,088.201
50 Acetone AC	0.00	0.000	271	0
50 Acetone AC	15.08	1.410	256	4,971.686

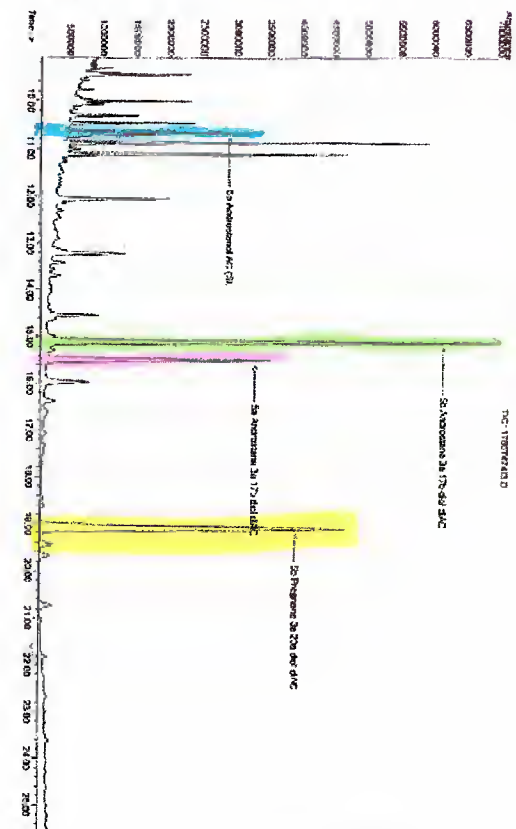
EX. 24, USADA 0172, 0173

EX. 24, USADA 0171, 0144

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E:\P
Operator      123456789010610100747473.D
Acquired      123456789010610100747473.D
Instrument     123456789010610100747473.D
Sample Name    123456789010610100747473.D
Misc Info      123456789010610100747473.D
Well Number    123456789010610100747473.D

```

[illegible]

Naïve	Ref. Est.	Ref. Est. Time	TauEst Speed	TauEst Response
Small molecule AC	0.07	0.000	256	4,350,622
Ethanolamine AC	0.00	0.272	272	0
Aminoacetic AC	0.00	0.000	272	0
5s Aminoalan 3s 17s dtd diac	15.14	1.418	256	15,590,168
5s Aminoalan 3s 17s dtd diac	15.50	1.452	256	8,733,825
11 Ketocyclopentanone AC	0.00	0.000	272	0
11 Ketocyclopentanone AC	15.72	1.372	274	20,400,363

EX. 25, USADA 349, 350

EX. 25, USADA 348, 321

LNDD	ENREGISTREMENT	Codification : E-FCR-06
		Version : E
		Date : 24/11/05
Page : 1/2		
FICHE D'ANALYSE / RESULTATS GC/C/IRMS		

Echantillon : 178/07 995474

Instrument : GC/C/IRMS Isoprime 1

Répertoire : 230706

CO et paraphe :

49

Valeur isotopique du réactif de dérivation :

-53

Fraction F1 (métabolites de la cortisone et du cortisol)

	Blanc urinaire		Echantillon	
	SI	11 Kétoétio	SI	11 Kétoétio
Nom du fichier	Data_010	Data_010	Data_011	Data_011
tr (s)	867	1474	867	1478
trr	-	1.700	-	1.705
Intensité (nA)	3.7	3.3	4.0	4.6
$\delta^{13}\text{C}$ ‰ mesurée	-30.80	-24.55	-31.64	-24.10
$\delta^{13}\text{C}$ ‰ corrigée	-	-21.56	-	-21.06

Fraction F2 (Kétos)

	Blanc urinaire			Echantillon		
	SI	Etio	Andro	SI	Etio	Andro
Nom du fichier	Data_012	Data_012	Data_012	Data_013	Data_013	Data_013
tr (s)	868	1232	1257	866	1230	1254
trr	-	1.419	1.448	-	1.420	1.448
Intensité (nA)	2.7	4.5	5.3	2.2	4.0	3.4
$\delta^{13}\text{C}$ ‰ mesurée	-29.94	-25.34	-24.98	-30.07	-26.43	-27.71
$\delta^{13}\text{C}$ ‰ corrigée	-	-22.43	-22.03	-	-23.63	-25.05

Fraction F3 (Diols)

	Blanc urinaire			
	SI	5 β Adiol	5 α Adiol	5 β Pdiol
Nom du fichier	Data_008	Data_008	Data_008	Data_008
tr (s)	867	1306	1337	1652
trr	-	1.506	1.541	1.904
Intensité (nA)	6.2	7.1	2.3	3.6
$\delta^{13}\text{C}$ ‰ mesurée	-30.66	-27.54	-28.40	-26.65
$\delta^{13}\text{C}$ ‰ corrigée	-	-22.18	-23.22	-21.63

	Echantillon			
	SI	5 β Adiol	5 α Adiol	5 β Pdiol
Nom du fichier	Data_009	Data_009	Data_009	Data_009
tr (s)	867	1305	1337	1652
trr	-	1.504	1.542	1.905
Intensité (nA)	6.8	5.5	2.6	3.3
$\delta^{13}\text{C}$ ‰ mesurée	-30.05	-28.82	-32.12	-26.61
$\delta^{13}\text{C}$ ‰ corrigée	-	-23.73	-27.72	-21.58

USADA 0185

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Credibility

“But in particular, as this case has progressed, to this point it has become truly about the credibility and the integrity of LNDD and its procedures and bluntly, LNDD’s and USADA’s attempts to cover up the errors that LNDD committed.”

Appellant's Defense Strategy

“He was unaware of what was going on,” Maurice said when he returned.

“So I explained that our defense was essentially to take down the French lab in an embarrassing way.”

-Positively False, p. 275